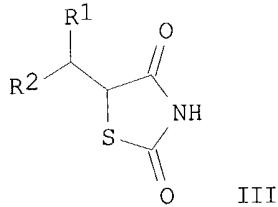
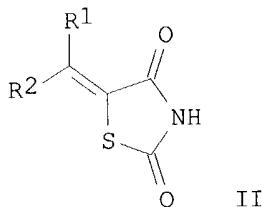


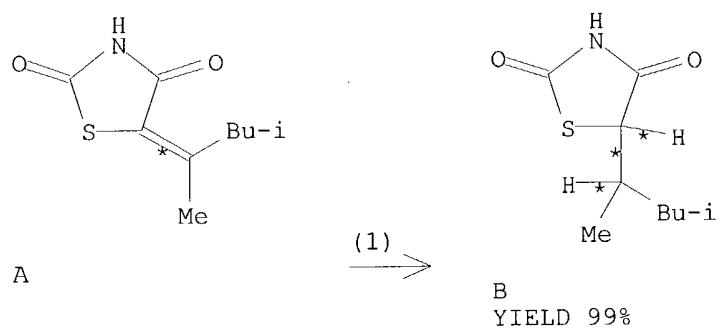
ACCESSION NUMBER: 134:100561 CASREACT  
 TITLE: Method for preparation of .alpha.-mercapto carboxylic acid  
 INVENTOR(S): Takahashi, Hiroshi; Ishii, Seiichi; Yoshida, Kei;  
 Tomiya, Kanji; Kodaka, Kenji  
 PATENT ASSIGNEE(S): Mitsui Chemical Industry Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001011042	A2	20010116	JP 1999-187566	19990701
PRIORITY APPLN. INFO.:			JP 1999-187566	19990701
OTHER SOURCE(S):		MARPAT 134:100561		
GI				



AB .alpha.-Mercapto carboxylic acids represented by formula R1R2CHCH(SH)CO2H [II; R1, R2 = C1-6 alkyl or R1R2C = (un)substituted C5-8 cycloalkane] are prepd. by condensation of thiazolidine-2,4-dione with R1R2CO (R1, R2 = same as above) with NH3(g), primary alkylamine, or amine carbonate, bicarbonate, or acetate salts to obtain 5-alkylidenethiazolidine-2,4-diones (II; R1, R2 = same as above) and catalytic hydrogenation of II to 5-alkylthiazolidine-2,4-diones (III; R1, R2 = same as above) followed by hydrolysis to I. I are useful as intermediates for drugs or agrochems., e.g. fungicidal 2-alkyl-3-aminothiophenes. Thus, Me iso-Bu ketone was condensed with thiazolidine-2,4-dione in the presence of 0.82 g ammonium carbonate and 7.20 g MgSO4 at 90.degree. for 11 h to give 79.8% 5-(1,3-dimethylbutylidene)thiazolidine-2,4-dione (98.5% purity) which was hydrogenated over 5% Pd-C in AcOH at 40 kg/cm2 and 80.degree. for 6 h to give 99% 5-(1,3-dimethylbutyl)thiazolidine-2,4-dione. The latter compd. (15.0 g) was mixed with 93 mL H2O, followed by blowing N, which was passed through blue silica gel, into the mixt. for 1 h, and 14.9 g NaOH was added in one portion and heated at 80.degree. for 8 h, cooled to room temp., and extd. with Et2O. The aq. layer was adjusted to pH 1.0 with concd. HCl and extd. twice with Et2O and the combined ext. was dried over MgSO4, filtered, and concd. to give 81% 3,5-dimethyl-2-mercaptohexanoic acid.

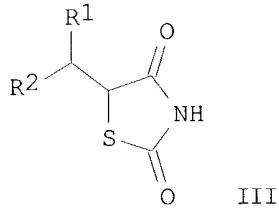
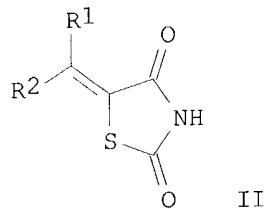
RX(1) OF 6      ...A ==> B...



RX(1)      RCT A **98959-96-7**  
              RGT C 1333-74-0 H2  
              PRO B **320350-17-2**  
              CAT 7440-05-3 Pd  
              SOL 64-19-7 AcOH

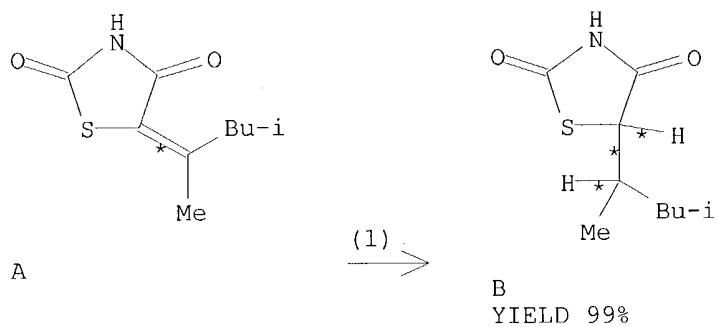
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AB .alpha.-Mercapto carboxylic acids represented by formula R<sub>1</sub>R<sub>2</sub>CHCH(SH)CO<sub>2</sub>H [I; R<sub>1</sub>, R<sub>2</sub> = C<sub>1</sub>-6 alkyl or R<sub>1</sub>R<sub>2</sub>C = (un)substituted C<sub>5</sub>-8 cycloalkane] are prep'd. by condensation of thiazolidine-2,4-dione with R<sub>1</sub>R<sub>2</sub>CO (R<sub>1</sub>, R<sub>2</sub> = same as above) with NH<sub>3</sub>(g), primary alkylamine, or amine carbonate, bicarbonate, or acetate salts to obtain 5-alkylidenethiazolidine-2,4-diones (II; R<sub>1</sub>, R<sub>2</sub> = same as above) and catalytic hydrogenation of II to 5-alkylthiazolidine-2,4-diones (III; R<sub>1</sub>, R<sub>2</sub> = same as above) followed by hydrolysis to I. I are useful as intermediates for drugs or agrochems., e.g. fungicidal 2-alkyl-3-aminothiophenes. Thus, Me iso-Bu ketone was condensed with thiazolidine-2,4-dione in the presence of 0.82 g ammonium carbonate and 7.20 g MgSO<sub>4</sub> at 90.degree. for 11 h to give 79.8% 5-(1,3-dimethylbutylidene)thiazolidine-2,4-dione (98.5% purity) which was hydrogenated over 5% Pd-C in AcOH at 40 kg/cm<sup>2</sup> and 80.degree. for 6 h to give 99% 5-(1,3-dimethylbutyl)thiazolidine-2,4-dione. The latter compd. (15.0 g) was mixed with 93 mL H<sub>2</sub>O, followed by blowing N, which was passed through blue silica gel, into the mixt. for 1 h, and 14.9 g NaOH was added in one portion and heated at 80.degree. for 8 h, cooled to room temp., and extd. with Et<sub>2</sub>O. The aq. layer was adjusted to pH 1.0 with concd. HCl and extd. twice with Et<sub>2</sub>O and the combined ext. was dried over MgSO<sub>4</sub>, filtered, and concd. to give 81% 3,5-dimethyl-2-mercaptophexanoic acid.

RX(1) OF 6      ...A ==> B...



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